



Research paper

Stability of kaolin dispersion in the presence of lignin-acrylamide polymer

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ABSTRACT

Dispersion of kaolin in aqueous systems is important in many industries including ceramics, chemicals, pharmaceuticals and paints. In this work, kraft lignin-acrylamide (KAM) polymers were produced via polymerizing kraft lignin and acrylamide (AM) monomer with different molar masses and charge densities, and they were used for stabilizing kaolin dispersion at varied pHs. The surface tension and contact angle studies demonstrated that KAM improved the surface hydrophilicity of kaolin mineral particles. Among KAM polymers (KAM-1, KAM-2 and KAM-3), KAM-3 with the highest molar mass (M_w) of 97,000 g/mol and charge density of -2.1 meq/g had the highest adsorption of 2.16 mg/g onto kaolin mineral particles. It was also found that KAM adsorption on kaolin mineral particles was pH dependent with KAM adsorption decreasing with increasing pH from 4 to 10. Generally, increasing ionic strength enhanced the adsorption of KAM on kaolin mineral particles. Salt reduced electrostatic repulsion between the anionic KAM polymers and the negatively charged kaolin mineral particles and facilitated the adsorption. Relationship between zeta potential and relative turbidity of kaolin dispersion was developed at different levels of KAM adsorption. KAM (especially KAM-3) increased the volume fraction of kaolin mineral particles in the dispersion. It also improved the stability of kaolin dispersion, and the impact was more noticeable at pH 10. Treating the dispersion with KAM was more effective than mechanical stirring in stabilizing kaolin mineral particles.

1. Introduction

The stabilization of aqueous kaolin dispersion is critical in the production of cosmetics, ceramics, paints, coating formulas and construction materials in order to obtain stable and homogenous colloidal systems (Brady et al., 1996; Zaman et al., 2002). Dispersants have been widely used in the stabilization of kaolin mineral particles via inducing electrostatic/steric repulsion forces between particles (Yuan et al., 1998; Boisvert et al., 2001). As kaolin minerals carry slightly negative charges, anionic polymers are normally the most efficient dispersants for kaolin dispersion (Das and Somasundaran, 2001; Konduri and Fatehi, 2017).

The properties of dispersants play key roles in stabilization of dispersion systems. In the past, the utilization of carboxyl containing synthetic polymers, e.g., polyacrylamide (PAM), for stabilizing kaolin dispersion was reported (Agnes et al., 1981; Nabzar et al., 1987; Pefferkorn et al., 1987; Lee and Somasundaran, 1989; Morris et al., 2002; Nsib et al., 2006; Kim and Palomino, 2009; Aso et al., 2013; He and Fatehi, 2015; Konduri et al., 2015). However, synthetic polymers may be expensive and environmentally unfriendly. Recently, reports were made available on the modification of lignin, which is a natural polymer extracted from wood via mainly pulping processes, to produce

semi-synthetic polymers with cationic and anionic charge densities (He and Fatehi, 2015; Couch et al., 2016; He et al., 2016, 2017; Konduri and Fatehi, 2017). The polymerization of lignin and acrylamide can lead to a polymer with more environmentally friendly features than PAM. As this polymer had a negative charge density, it would function as a dispersant for kaolin dispersion. In this regard, lignin-acrylamide polymers with different molar masses and/or charge densities can be produced. These polymers have different physical and chemical characteristics than synthetic polymers, which may impact their dispersion performance (Pefferkorn et al., 1987; Rong et al., 2013a,b). The first objective of this work is to study the impact of lignin-acrylamide (KAM) polymer as a dispersant for kaolin dispersion.

The behavior of kaolin mineral particles in the dispersion is strongly affected by the chemistry of the dispersion, such as dispersion pH and salt concentration (Nsib et al., 2006; Chibowski et al., 2009). Variations in pH were also reported to alter the adsorption of polymers onto kaolin mineral particles (Atesok et al., 1988). Similarly, salt significantly affected the interaction of particles in dispersions, and thus the interaction and performance of dispersants in dispersion systems (Shirazi et al., 2003; Chibowski et al., 2009; Mishra et al., 2014). The second objective of this work is to investigate how pH and salt in kaolin dispersion impact the performance of lignin-acrylamide polymer as a

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dispersant.

The main novelty of this study is the investigation of the dispersion performance of kraft lignin-acrylamide polymer in kaolin dispersion at different pHs and salt concentrations. The correlations between KAM adsorption on kaolin mineral particles, and the changes induced by KAM on the zeta potential, relative turbidity, particle size and stability of kaolin minerals in dispersion are established for the first time.

2. Experimental

2.1. Materials

Softwood kraft lignin (KL) was produced via LignoForce™ technology of FPIInnovations in Thunder Bay, ON (Kouisni et al., 2012). Acrylamide, AM (99.0 wt%), potassium persulfate ($K_2S_2O_8$) (analytical grades), potassium hydroxide, para-hydroxybenzoic acid, hydrochloric acid (0.1 M), $NaNO_3$, NaOH, H_2SO_4 (98%), KCl, NaCl (all analytical grades) and kaolin were all obtained from Sigma-Aldrich company. Cellulose acetate dialysis membrane (molar mass cut off of 1000 g/mol) was obtained from Spectrum Labs. Inc., USA. All chemicals were used without further purification. Ethanol (95 vol%) was received from Fisher Scientific company. Potassium polyvinyl sulfate (PVSK) was provided by Wako Pure Chem. Ltd. Japan. Polydiallyldimethyl ammonium chloride (PDADMAC) with the molar mass of 100,000–200,000 g/mol was obtained from Sigma Aldrich company and diluted to 0.005 M prior to use.

2.2. Polymerization of lignin

The free radical polymerization of KL was carried out in a 250 mL three-neck round-bottom glass flask under the reaction conditions listed in Table 1. First, a required amount of KL was dissolved in 40 mL of deionized water while stirring at 300 rpm. Predetermined quantities of AM were added to the flask and stirred for 30 min. The pH of the solution was adjusted using 0.1 M sulfuric acid. The reaction solution was continuously purged with nitrogen to remove any residual oxygen at room temperature for 30 min. Subsequently, potassium persulfate was added as an initiator to the system and the reaction solution was purged for another 5 min. The polymerization was processed by placing the flask in a preheated water bath after adjusting the pH of the system. The reaction was allowed to proceed for the desired time intervals under a continuous nitrogen supply. These reaction conditions generated lignin-acrylamide polymers with a high molar mass, charge density and solubility in a previous study (Wang et al., 2016a).

After completion of reaction, the solution was cooled to room temperature by immersing the flask in tap water for 20 min. The kraft lignin-acrylamide polymer was precipitated by adjusting the solution pH to 1.5 using sulfuric acid. Then, the sample was centrifuged at 3500 rpm for 10 min using a Sorvall ST 16 laboratory centrifuge (Thermo Fisher) in order to separate the lignin polymers from

homopolymers (polyacrylamide, PAM) and unreacted acrylamide monomers. This precipitation/centrifugation process was repeated three times and then the lignin-acrylamide polymers were collected. Then, the precipitated lignin polymers were mixed with 200 mL of deionized water. After adjusting the pH of the solution to 7.0 using a 1 M NaOH solution, the samples were dialyzed using the dialysis membrane for 48 h in order to remove impurities (e.g., inorganic salts and monomers) from the polymer solutions. The deionized water used for dialysis was changed every 12 h for 2 days. After dialysis, the solution containing lignin-acrylamide samples were dried at 105 °C, and the dried samples were kept for further analysis. This lignin polymer is denoted as KAM, while unmodified kraft lignin is denoted as KL in this work. All experiments were repeated three times, and the average data and error bars are presented in this work.

2.3. Specific surface area analysis of kaolin

The specific surface area of kaolin was determined by using Quantachrome surface area analyzer, a Nova 2200e instrument, USA. In this experiment, the samples were initially dried in an oven at 105 °C overnight, and approximately 0.05 g of sample was pretreated for 4 h at 250 °C prior to analysis. The specific surface area of the samples was then determined according to Brunauer-Emmett-Teller (BET) method via adsorption-desorption isotherms using nitrogen gas at -180 °C in the relative pressure range of 0.01 and 0.99 (Oveissi and Fatehi, 2014).

2.4. Chemical analysis of kaolin

The chemical composition of kaolin was determined using scanning electron microscopy (SEM), Hitachi Su-70, with energy dispersive X-ray spectroscopy (EDX) (Senoussi et al., 2016). In this set of experiments, 0.2 g of kaolin was dispersed in 20 mL of acetone under ultrasonic vibrations for 5 min and allowed to air dry for 24 h. After drying, the sample was coated with carbon using Edward Auto 306 system (Edward International Corp, UK) under vacuum (3×10^{-5} mbar) for 10 min prior to SEM analysis.

X ray diffractometry (XRD) analysis was carried out to determine the compositions of the samples and their distribution using spinning stage PANalytical X'pert-PRO diffractometer (XRD), PW1050–3710 with a $Cu K\alpha$ ($\lambda = 1.5405 \text{ \AA}$) radiation source (Senoussi et al., 2016). A 0.5 g of kaolin was air dried and transferred onto the spinner of XRD. The XRD scan of kaolin was performed in a continuous mode from 6 to 97° with a 0.026° step size and a scan speed of 0.164 (°/Sec) for total scan time of 9 min. The chemical compositions were identified by analyzing the positions of the peaks using the X'pert High Score software package supplied with the instrument (Mcintosh et al., 2015). The loss on ignition (LOI) of kaolin was determined via following TAPPI T413 method by incinerating the kaolin at 900 °C for 8 h (Santisteban et al., 2004).

Table 1
Reaction conditions and properties of KAM.

| Conditions | KL | KAM-1 | KAM-2 | KAM-3 |
|-------------------------------|----------------|----------------|----------------|----------------|
| Temperature, °C | – | 90 ± 0.02 | 60 ± 0.03 | 80 ± 0.02 |
| Time, h | – | 5 ± 0.03 | 2 ± 0.03 | 2 ± 0.02 |
| KL, mol | – | 0.016 ± 0.01 | 0.011 ± 0.01 | 0.016 ± 0.01 |
| AM, mol | – | 0.014 ± 0.01 | 0.014 ± 0.01 | 0.042 ± 0.01 |
| pH | – | 4 ± 0.2 | 2 ± 0.2 | 5 ± 0.22 |
| Initiator, mmol | – | 0.11 ± 0.01 | 0.11 ± 0.01 | 0.11 ± 0.01 |
| Anionic charge density, meq/g | 0.21 ± 0.03 | 0.82 ± 0.03 | 1.37 ± 0.05 | 2.1 ± 0.02 |
| COOH content, mmol/g | 0.14 ± 0.01 | 0.63 ± 0.02 | 1.1 ± 0.03 | 1.78 ± 0.04 |
| Solubility, g/L | 0.5 ± 0.04 | 4.5 ± 0.1 | 4.5 ± 0.1 | 4.7 ± 0.09 |
| M_w , g/mol | 17,890 ± 11.54 | 24,590 ± 10.34 | 50,215 ± 11.76 | 96,992 ± 10.23 |
| M_n , g/mol | 5150 ± 5.77 | 23,129 ± 9.77 | 28,741 ± 8.55 | 69,716 ± 10.35 |
| Nitrogen, wt% | 0.03 ± 0.01 | 1.57 ± 0.03 | 3.8 ± 0.05 | 6.2 ± 0.05 |

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